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asbestos analysis is dependent upon the skill and experience of the analyst, aided by sophisticated equipment. Although the basic techniques of identification and counting can be learned fairly quickly on ideal samples, only experience and closely supervised training can produce reliable results with real samples.

NVLAP requires that each laboratory provide a written training program for TEM analysts, and what is presented here is the outline of a basic training program, including some of the proposed NVLAP requirements.

ANALYST QUALIFICATIONS

People who have been involved with setting up and teaching TEM training courses usually agree on the items which need to be covered and the general order in which they should be taught. One of the difficulties, however, is matching the level of training with the analysts' background skills. A company must invest a great deal of time and money in training good analysts, and must continue to train them for the constantly changing requirements we see in the industry today. The beginning qualifications of the analyst-in-training are, therefore, important.

Hiring criteria for TEM analysts usually focus on the following items. The educational level of the person is the first item of interest. Typically, an analyst will have at least a bachelor's degree completed or in progress, usually in one of the sciences. This, we hope, suggests clear communication skills and an interest in the analytical aspects of the work. Geology or materials science majors are desirable, but not required.

Graduate level work is a plus, particularly if the work includes training in areas related to the analysis. Knowledge of electron microscopy, either scanning or transmission, x-ray diffraction, electron diffraction or energy dispersive x-ray spectroscopy will make the training easier, and of course knowledge of mineralogy, crystallography and polarized light microscopy are an asset. One of the most common deficiencies in beginning analysts is a complete lack of knowledge about the asbestos industry that they are joining, and for this reason people who have some experience in environmental health, asbestos industrial hygiene or field work are valuable, as they are able to deal well with clients and can often recognize obvious inconsisten-

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cies in samples. We should also remember that highly qualified people can be trained more quickly, but may not be satisfied with routine analytical work over the long run.

OUTSIDE TRAINING

Assuming that an analyst can be found who has an acceptable level of knowledge, we should first examine the education and training that is available outside the laboratory.

Obviously the most specific training is one of the recognized TEM asbestos analysis courses being taught around the country. Typically, these consist of one week of general TEM training and a second week which focuses on specific asbestos analysis techniques and current requirements, particularly for AHERA. The best way to assess these courses is to talk to other lab people who have attended them.

There are also a number of advanced courses being offered such as analytical electron microscopy, advanced electron diffraction and energy dispersive x-ray spectroscopy. The equipment manufacturers all offer orientation and basic user courses for their products. Professional development seminars are excellent training tools, as are national and regional conferences of professional organizations which many analysts join.

Outside courses and meetings help to increase communication about analysis techniques and potential problems; this makes our analyses more consistent and reliable.

IN-HOUSE TRAINING

Turning to the in-house training program, which will be the cornerstone of the analyst's training, we see that there are many aspects that must be addressed before the analyst starts accumulating microscope time.

Every lab should have a library of written training materials and new analysts must become thoroughly conversant with them. Safety and health information should come first, covering the dangers of handling asbestos, working with chemicals and equipment in the lab, exposure to x-rays from the electron microscope and radiation badge monitoring.

The trainee should study the *Quality Assurance Manual* to achieve an idea of the overall quality system requirements and procedures.

The basic analysis methods should be read early in order to understand the goal the training is designed to reach. Obviously, the AHERA document(1), the Environmental Protection Agency (EPA) draft air method(2), the National Institute of Occupational Safety and Health NIOSH 7402 method(3) and Burdett and Rood paper(4) are minimum requirements, and other asbestos analysis methods(5) should be included as needed.

The equipment manuals and training information, such as tapes and filmstrips, should also be studied before the equipment is used.

The training library should include relevant texts on electron microscopy, energy dispersive x-ray spectroscopy, electron diffraction, mineralogy, crystallography, asbestos mineral characteristics and identification and other subjects. Important journal articles and government publications related to asbestos analysis and regulation should be added to the library regularly. There is a large amount of research in progress concerning analysis techniques and sampling methodology, that all analysts should be aware of.

NVLAP requires that Standard Operating Procedures (SOP) be written up by each lab for a variety of items. Basically, if you must explain to someone how a procedure or analysis is done, you should write up an SOP. This insures that everyone starts with the same information, and greatly speeds up the training process.

Ideally, new analysts should be apprenticed to a single experienced analyst, but, more typically, the training is shared by several analysts. The training should include demonstrations, hands-on work, testing and documentation.

Some of the items requiring SOP's include sample receiving, handling, custody, storage and disposal procedures, which should be familiar to everyone.

Some prep methods and clean room procedures, particularly contamination control, are another important SOP topic.

The requirements and training for sample prep personnel will not be addressed here, but even in large labs it is sometimes necessary for analysts to prep samples, and they should know the procedures. In addition, this knowledge allows analysts to recognize prep problems and communicate with prep personnel for better sample preparation.

SOP's for the use of equipment such as plasma ashers and

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carbon coaters should be available, as well as maintenance information on all equipment.

The electron microscope itself should have SOP's for basic use, in addition to the manufacturer's instructions. Items to be covered include beam alignment procedures for maximizing image formation and electron diffraction patterns and minimizing astigmatism, sample damage and other problems. At this point, the analyst can begin hands-on training with the TEM, under supervision of an experienced analyst. SOP's and demonstrations should be used to teach the proper methods of magnification and camera constant calibration(6), set up, calibration and operation of the energy dispersive x-ray spectroscopy system and all other aspects of daily TEM operation.

Only after the experienced analyst is satisfied that the trainee is properly acquainted with TEM operation should the trainee begin asbestos analysis techniques.

NVLAP specifically states that each lab must write procedures for all aspects of the asbestos analysis and handling, not just use a copy of the method. This apparently means that an analysis SOP must be provided, reiterating the methods of identification and counting as interpreted by the lab. Following such procedures, the trainee can now begin to learn specific asbestos analysis techniques. AHERA, EPA methods such as Yamate Level II and other procedures should be covered.

SOP's for data handling and calculations must also be provided, as well as requirements for documenting items in the quality system.

Electron diffraction training is an extremely important part of an asbestos analyst's training, and can be a very complicated and time-consuming thing to teach. It is in this area that analysts with some geology background often have an advantage. If the trainee is not familiar with basic crystallography and mineralogy concepts, then this must first be covered. The training should get into an understanding of crystal structure, Miller indices, reciprocal space, the Ewald sphere, etc. The characteristic chrysotile pattern should be understood completely, as this pattern is actually formed from a number of zone axis patterns in the rolled chrysotile fiber. Amphibole zone axis patterns should be taught in detail, and indexing and identification of electron diffraction patterns practiced.

The use of stereographic projections and Wulff nets to plot

inter-planar and inter-zonal angles must be covered as part of the more advanced aspects of diffraction.

Actual TEM work with diffraction should include training and practice with either the double tilt or tilt/rotate sample holder which every lab should have. Proper procedures for photographing diffraction patterns, recording tilt angles and fiber orientation and developing the EM film properly must be taught along with how to document the findings(7).

Once the trainee is familiar with the concepts and procedures of electron diffraction, he should take the time to examine samples of the asbestos minerals, note their similarities and differences and photograph zone axis patterns. Acquiring such patterns is a skill which comes very slowly at first, but becomes more routine with sufficient practice. Since chrysotile is the asbestos type most commonly encountered, the analysts should be trained to identify the diagnostic diffraction pattern quickly on the screen, as well as from a photograph, as the pattern may only be visible briefly, or may be complicated by other material near the fiber.

Typical non-asbestos materials should be examined closely. In particular, gypsum, halloysite, palygorskite or attapulgite, sepiolite, talc, wollastonite, vermiculite and hornblende should be examined. All of these minerals have the potential to produce structures which meet the EPA or AHERA TEM fiber-size definition, and many give diffraction patterns which can be confused with a chrysotile or amphibole pattern by an inexperienced analyst. Some of these minerals appear in air samples with regularity, while others are rare but can occur, particularly in water or other sample types. Man-made fibers such as mineral, rock and slag wool, ceramic and graphite fibers and fiberglass must also be included in the non-asbestos category.

Luckily, most of the problem fibers we see are easily eliminated by energy-dispersive x-ray spectroscopy analysis, known as energy dispersive spectroscopy (EDS) or energy dispersive x-ray analysis (EDXRA). The trainee should therefore examine the EDS spectra of the asbestos and non-asbestos minerals when he does diffraction training. He should be familiar with the set-up and operation of the EDS unit, particularly the calibration tests that NVLAP requires. These include calibration of resolution and sensitivity using the National Institute for Standards and Technology, (NIST) Standard Reference Material (SRM) 2063

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thin film standard, which contains the elements important to asbestos analysis, *i.e.*, sodium, magnesium, silicon, calcium and iron. This is a new standard, and must be purchased from NIST for NVLAP work. In addition, the analyst should be aware of the ability of the EDS analyzer to resolve the sodium peak in a crocidolite fiber and acquire usable magnesium and silicon ratios on a single fibril of chrysotile. The analyst should report any deficiencies in the equipment if noticed.

One of the most common problems encountered with new analysts using EDS is the tendency to blindly accept the spectrum readout on the machine without considering interferences. Asbestos fibers can be coated with other materials or lie in such close proximity to non-asbestos material that even use of a very small spot size will not prevent the acquisition of extra elements. Analysts should be taught to be suspicious of EDS spectra, and to analyze several areas of the fiber or nearby material to detect interferences. Electron diffraction should always be examined before EDS is used, of course, as beam damage by EDS often renders diffraction pattern formation impossible, especially on small chrysotile fibers.

When the analyst is comfortable with the basic TEM operation, diffraction and EDS, he can start training and testing procedures on a variety of samples.

As mentioned, asbestos standard materials and non-asbestos materials of interest should be studied. NVLAP also requires characterization of the SRM 1866 bulk sample materials, which include chrysotile, amosite, crocidolite and fiberglass.

The counting procedures(8) for AHERA and EPA methods must be practiced and learned on a variety of samples. The NIST SRM 1876 and reference materials (RM) 8410 and 8411 chrysotile standards have a documented concentration of asbestos structures per square millimeter, and should be used for training and testing. Additional mixtures of chrysotile, amphiboles and non-asbestos fibers should be made up in the laboratory, for counting and identification practice.

Quality Control

When the trainee is comfortable with standard materials, it is time to try Quality Control (QC) analyses of client samples that have been analyzed by experienced analysts. They should be

selected for a wide variety of structure types, loadings and difficult interfering materials. An experienced analyst should be available to help the trainee, and results can be compared using Poisson or normal statistics, as appropriate.

At this point in the analyst's training, he should also be familiar enough with the equipment to undergo hands-on testing or "practicums." This consists of deliberate misalignment or creation of other problems by an experienced analyst, which the trainee must identify and correct. This has been shown to be a very good way of teaching equipment use.

The NVLAP handbook includes a training and testing procedure developed at NIST, which, if properly used, is a very worthwhile tool. This is the Verified Asbestos Analysis (VAA); it is explained in the NVLAP handbook and in two papers referenced there.

Briefly, the procedure involves counting of the same grid squares on a standard or real sample by multiple analysts. The asbestos structures are identified, sized and a description or drawing of each structure is made. This allows the analysts to compare structures and reveals problems in identification or counting rules interpretation. If an analyst is missing structures by scanning too rapidly or not leaving enough screen overlap between passes, this will be immediately apparent. This procedure can be done within the laboratory and with interlab exchange samples, as well as on the NVLAP round-robin proficiency samples. The samples used for this should have 1000 to 5000 structures per square millimeter, or approximately 8 to 40 structures per 200 mesh TEM grid opening. Analysts should score greater than 80% true positives (less than or equal to 20% false negatives) and less than 10% false positives. The lab should have at least one highly qualified analyst, usually the lab manager or supervisor, with a record of greater than 85% true positives and less than 5% false positives. Most labs will require better performance than this, but as a working level this is sufficient. Experience with this verified analysis soon shows that problems with identification and missed fibers are not as common as differences in counting rules interpretation. For example, an analyst who counted three fibers as a cluster could easily exceed the 85% rule on a lightly loaded grid opening when compared with the analyst who counted the structure as three individual fibers. The counting rules are vague enough that

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unusual asbestos structures are open to subjective interpretation(8). A result of the verified analysis procedure is that analysts in the same lab will start to agree with each other more closely as the counting rules are agreed on. We should remember, however, that another lab may be interpreting the rules somewhat differently. It is hoped that any verified asbestos analyses performed by NIST would include a written critique of structure counts, as well as the return of the sample grid so that the lab can recheck its findings. The biggest practical problem with this analysis is that use of SAED and EDS for identification will render chrysotile fibers non-diffracting very quickly. For this reason, it is useful to do this analysis using SAED only for identification, or even using morphology only for large numbers of analysts, especially if the training is for counting rules practice. NVLAP considers the verified analysis to be an important part of the training procedure as well as documentation of each analyst's skill.

The exercise of quality control over a number of TEM analysts' output requires constant supervision and attention. New analysts in particular are liable to feel, at some point, that they are fully trained and may resent continual checking of their work, but the experienced analysts must continue to assess the trainees' results. It is particularly important that all analysts feel comfortable with asking questions and seeking assistance if there is any doubt about a sample result.

Decreasing quality control checks can be initiated when a new analyst is ready to analyze actual samples. After successful training at the 100% re-analysis level, a new analyst can be dropped to a 50% level, with close supervision. This usually involves two analysts, each analyzing one-half of the total number of grid openings and then comparing fiber counts and notes on sample quality. It is very helpful if the analysts briefly note the overall sample loading and the presence of typical non-asbestos fibers. For example, the notation: "moderate particle loading, gypsum and aluminum-silicate fibers common" by a trainee, assures the QC analyst that the trainee has taken the time to observe particle distribution and verify the identity of suspicious fibers.

After a trainee has successfully demonstrated that he is capable of meeting the 50% QC checks, he can be dropped to a 10% QC check level, which involves a QC analyst completing

the last grid opening in 10, or some similar scheme. This level of QC is continued for all analysts in our lab at least every third sample. It is particularly useful, since it is a "productive" quality control check, being incorporated into the actual analysis. Poisson or normal statistics may be used to compare the primary and QC analysts' results, and analysts are encouraged to discuss problems encountered in the sample. This QC method is particularly good for small sample batches, which might otherwise escape a QC check, and lets new analysts know that they are not working alone.

It is also recommended that analysts print-out the EDS spectrum of a typical asbestos structure of each type found in each sample. EDS spectra of suspicious fibers should also be printed out. These printouts can be included with the raw data sheet, which should, of course, be reviewed by an experienced analyst before calculations and client reporting. The problem of electron diffraction photographs is more complicated, especially with ever-decreasing sample turn-around times. Analysts should follow AHERA or NVLAP guidelines as a minimum requirement, and new analysts should, of course, request assistance with zone axis amphibole patterns until they have demonstrated the ability to properly acquire and index these. On the other hand, visual identification of chrysotile diffraction patterns is critical. Analysts who are not confident in their ability to do this will consistently underestimate fiber concentrations, compared to experienced analysts.

Proficiency Testing

After new analysts have reached full-training level, they must participate in all intra-analyst, inter-analyst, inter-laboratory and proficiency testing sample rounds, as required. NVLAP will require that all analysts analyze the proficiency samples, although only one result will be reported to NVLAP. The individual analyst's results will be kept in their file for review by the assessor.

NVLAP also mentions that the laboratory's overall degree of precision and accuracy must be reported, and this information is calculated from the individual analyst's VAA results. This requirement is currently on hold by NVLAP, however.

There are, of course, many additional training items in a

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TEM lab, and many depend on the size of the lab and job requirements for TEM analysts. In most labs, analysts must be capable of handling and calculating the data, although the main computer entry work may be handled by the support staff. The analyst must nonetheless provide data recalculation checks and be able to explain calculations to clients. For AHERA work, the rather confusing sequence of pass/fail steps must be fully understood and the Z-test calculations practiced.

Analysts will usually be trained to write client reports and handle questions, and they should be fully aware of client confidentiality rules.

As discussed before, analysts must be aware of the quality system review procedure, proper documentation and correction steps in the event of problems. The SOP's for such items should be clearly written and well understood.

As mentioned earlier, analyst training is an ongoing process, but documented, regular retraining is obviously required. On at least a yearly basis, analysts should be given time to review procedures and undergo retraining lectures.

If any evidence of misidentification or counting problems are revealed by the quality system checks, then the source of this must be located and corrected, of course. Asbestos analysis can become very tedious, and there is always a tendency for people to take shortcuts and to make assumptions based on previous experience. Any serious analyst problems require re-testing of skills before the analysts' results are again used for client samples, and this should be well documented.

Continuing Education

Ongoing training is the key to keeping analysts interested in their work and aware of problems. Regular laboratory meetings and seminars by staff members and guest speakers are recommended, and can be used as practice sessions for presentations to be given elsewhere. Quality system meetings are also important, and can be used to discuss comparisons of Verified Asbestos Analysis counts and other items. Professional Development Seminars given by groups such as the National Asbestos Council (NAC) are excellent training vehicles and people should be allowed at least one relevant conference per year, if the lab can afford this.

Staff members should be given every encouragement to do research and publications. This usually requires analysts to invest some of their off-hours, but most scientific people are aware of the career benefits of publications and will react favorably if encouraged by the company.

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